

EXTREME VACUUM TECHNOLOGY INCLUDING CRYOSORPTION,  
DIFFUSION PUMP AND PRESSURE CALIBRATION STUDIES

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by

Paul J. Bryant  
Charles M. Gosselin

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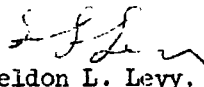
## PREFACE

The development of several phases of research relating to vacuum science and technology are included in this program. Mr. George Wise of the NASA Lewis Research Center is the technical monitor. This Quarterly Status Report covers the period from 1 February 1965 to 1 May 1965.

The research activity is conducted in the Physics Department of the Midwest Research Institute under the direction of Dr. Sheldon L. Levy and Mr. Gordon E. Gross. Research activities were conducted by Dr. Paul Bryant, Mr. Charles Gosselin and Dr. William W. Longley.

Approved for:

MIDWEST RESEARCH INSTITUTE

  
Sheldon L. Levy, Director  
Mathematics and Physics Division

28 May 1965

## SUMMARY

The research tasks of this program relate to the development or application of extreme vacuum science and technology. The tasks range from the production and calibration of extreme vacuum to the measurement of the rate of cryosorption of known gas species on clean surfaces.

The objectives of the phase which is reported in Section II were the determination of the residual gases present in an ultra-high vacuum system employing oil diffusion pumps and a study of the effects of various trapping conditions on the presence of oil contamination. For this study a special trap was designed which could employ either chemical or cryogenic trapping or it could operate with various combinations thereof.

The study was successful in two regards; first, hydrocarbon fractions in the UHV chamber were reduced to an exceptionally low level; and second, a useful combination of chemical trapping and moderate cryogenic temperature was discovered. This combination is as effective as a  $\text{LN}_2$  cooled metal trap alone and is especially practical, since the more moderate temperature ( $-78^\circ\text{C}$ ) can be maintained with mechanical refrigeration. An article describing this research phase has been submitted to the Journal of Vacuum Science and Technology and is included as Section II of this report.

Several other phases of research are being conducted simultaneously. (Each topic will be presented as a section of future reports when it has progressed to completion.) A brief summary of the remaining phases is given below.

A quartz piezoelectric crystal and oscillator have been designed and constructed for use as a micro-balance. The adsorption of helium gas on the crystal face at cryogenic temperatures below  $10^\circ\text{K}$  will be measured. The oscillator and quartz resonator assembly has been operated successfully with a quarter of a microwatt of power through a pair of 33 in. coaxial leads. (This feat had to be accomplished to permit data collection within an UHV system cryopump.) The operational stability of the system now indicates that a sensitivity of  $10^{-10}$  gm/cm<sup>2</sup> can be obtained (a monolayer of helium weighs  $10^{-8}$  gm/cm<sup>2</sup>), so that monolayer adsorption can be measured with one per cent accuracy.

A new field emission microscope tube has been designed in which the emitter tip can be cooled without significant cooling of the inner wall of the microscope tube. This feature will permit cryosorption of atoms onto the tip from a gas without altering the equilibrium pressure of that gas. Therefore, the number density of atoms may be studied without significantly influencing the total number. The geometry of this new microscope tube has been arranged so that a line of sight path is possible for gas molecules between each of the following three units: a nude Bayard-Alpert type ionization gauge, a nude source partial

pressure analyzer, and the microscope tip. That is, the three units are arranged in a line of sight within high conductance tubulation, so that they may all detect the same equilibrium gas pressure.

The vapor jet test apparatus was rebuilt so that more accurate data can be collected. Previous operation of a special dual expansion nozzle of the Fluorescu type revealed the need for higher sensitivity to adequately evaluate the nozzle operation. The redesigned system has been assembled and is now being operated.

A detailed study of the characteristics of magnetron vacuum gauge characteristics has been conducted. Further theoretical descriptions have been developed during the past three months and correlated with experimental data to provide an understanding of magnetron gauge response. Papers were presented on this subject at an American Vacuum Society seminar in Houston, Texas, and at the ASTM Conference on Mass Spectrometry in St. Louis, Missouri.

## I. INTRODUCTION

Vacuum science and technology has advanced considerably in the past few years. Some problems have been solved; however, new problems have been introduced. As is often the case, initial advances are made by the application of a new technique or process, but further advance requires an understanding of the process. There are now several areas of vacuum technology which require further development by means of an increased knowledge of the processes involved.

Several areas which require additional understanding are: the physical adsorption process in cryopumping; the vapor-jet pumping process; ultra-high vacuum measurements; and the type and origin of residual gas species in UHV systems. Each of these processes is being studied under this program.

## II. RESIDUAL GAS ANALYSIS OF A DC-705 OIL DIFFUSION PUMPED UHV SYSTEM\*

The residual gases present in a DC-705 oil diffusion pumped UHV system have been determined as a function of various trapping conditions. The system was equipped with a metal 2 in. oil diffusion pump in series with a specially designed chemical trap. The configuration of the trap permitted it to be immersed in cryogenic liquids, e.g.,  $\text{LN}_2$ . The effectiveness of the trap was determined as a function of temperature. A comparison of the residual gases is made for six conditions, including three trapping temperatures, while operating with chemicals and without chemicals.

### A. Introduction

Diffusion pumps have long been used to achieve high vacuum environments and in recent years UHV environments. The major advantage in using diffusion pumps is their capability to handle large gas loads for extended periods of time and to maintain pumping speed at reduced pressures. The limitations associated with the diffusion pumping technique are often established by the fluid used as a pumping media and the trapping technique used to isolate this fluid from the

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UHV chamber. Pressures well into the UHV range have been achieved using both mercury<sup>1,2/</sup> and oil<sup>3/</sup> as a pumping fluid in conjunction with optically dense liquid nitrogen baffles. Oil has also been used successfully with optically dense chemical traps at room temperature to obtain UHV pressures.<sup>4/</sup> However, in addition to the fact that a UHV can be obtained, it is important to know the type and quantities of residual gases for each pumping fluid and trapping combination.

A study was made to determine the residual gases present in an UHV system for various trapping conditions to aid in more carefully defining the contamination problem when using DC-705 pumping fluid.

#### B. Equipment

The apparatus used in this study can be divided into three major components, i.e., the partial pressure analyzer system (PPAS), the experimental trap, and the oil diffusion pump system.

1. The PPAS (shown in Fig. 1) consists of a G.E. Model 514 partial pressure analyzer (PPA), an 8 liter/sec getter ion pump, two closed grid ionization gauges, and the associated plumbing.

The analyzer unit (first described by Davis and Vanierslice<sup>5/</sup> and later by Davis<sup>6/</sup>) is a magnetic focusing device using a 5 cm. radius of curvature and a 90 degree sector. It is equipped with a Nier-Type ionization source and a ten-stage electron multiplier ion detector. A variable field strength electromagnet (0 to 6.5 kilogauss) is used with the PPA. Magnetic sweeping techniques are, therefore, employed in the collection of data.

An 8 liter/sec getter ion pump is connected to the analyzer system to provide an environment in which the PPA is maintained clean and in operating condition between data collection periods. A 1-1/2 in. isolation valve is installed between the analyzer and the getter ion pump to eliminate the effects of the active surfaces of the getter ion pump during the analysis of the test system.

Total pressure measurements are obtained by one of two glass ionization gauges. The other gauge is operated under a variety of electrical conditions to aid in determining the effects of ionization gauges on the residual gas spectra.

The remainder of the PPAS is constructed of 304 stainless steel and Kovar. Since all of the flange seals are made with copper gaskets, the entire PPAS (less electronic connection and magnet) is bakeable to 400°C.

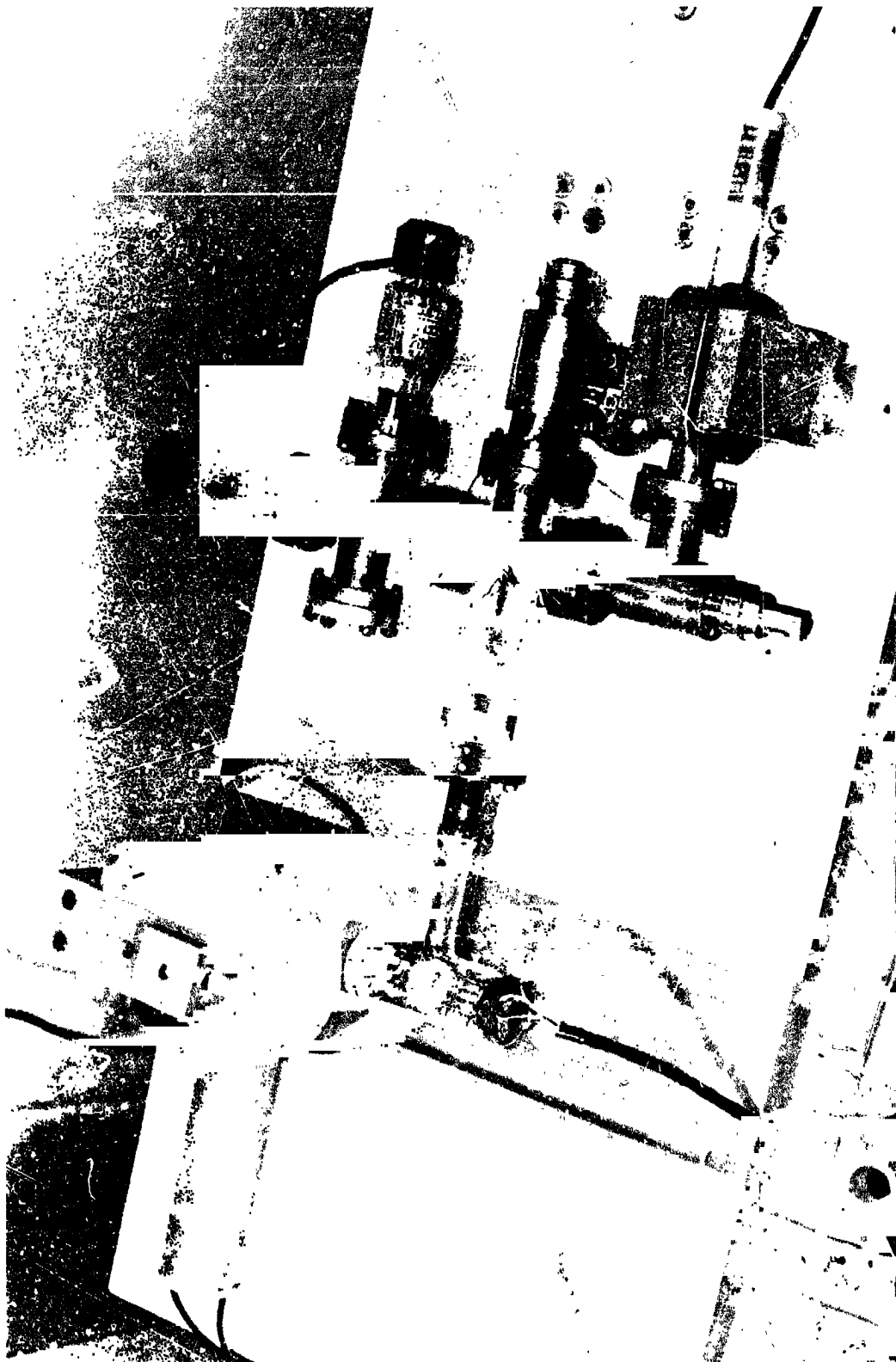


Fig. 1 - Partial Pressure Analyzer System (PPAS). Consist of a C. E. Model 514 analyzer (PPA) with a 6.5 kilogauss electromagnet, an 8 liters/sec getter ion pump, ionization gauges, and appropriate plumbing.



2. The experimental trap (shown in Fig. 2) is constructed of 304 stainless steel and is similar in design to the chemical trap proposed by Biondi.<sup>4/</sup> The trap is all heliarc welded, and thus fully bakeable. The design of the chemical zone or sump is such that it can be submerged in cryogenic fluids, e.g., liquid nitrogen or a dry ice and acetone slurry. Active chemicals such as CVC Absorbant-A are placed in the base of the sump and in the annular tray at the lower end of the vertical tube. This configuration is not only optically dense but also eliminates oil creep paths along walls of the trap (to the effective limit of the chemicals used).

3. The oil diffusion pump system (shown in Fig. 3) consists of a 2 in. pump backed by a 1 in. booster pump, a foreline valve, chemical foreline trap and a mechanical forepump. The 2 in. pump is trapped by a water-cooled chevron baffle. Gold O-ring seals are used between the 2 in. pump and baffle.

Copper gasket seals were used to connect the diffusion pump system, the experimental trap, and the PPAS. Therefore, the bakeable portion of the system extends from the top of the 2 in. diffusion pump to and including the PPAS.

### C. Results

A summary of the data collected is presented in Table I. Each column lists the air equivalent pressure for each gas species found in the system during the various conditions.

The background spectrum (first column of data in Table I) was recorded when only the PPA and one of the ion gauges were operating. That is, the analyzer had been sealed off from the diffusion pump, the chemical trap and the getter ion pump by closing the two isolation valves. The pressure remained at  $\sim 5 \times 10^{-10}$  torr for several days under these conditions.

The gas species which constituted the major portion of the background pressure were hydrogen and methane. It is interesting to note that a significant decrease in methane occurred when either the diffusion pump or the getter ion pump action was introduced. Following the evaluation of the chemical trap, a preliminary study was undertaken to determine the origin of methane in the PPAS. The results are reported at the end of this section.

The background spectra also indicate reasonably large amounts of carbon monoxide and helium. It has been observed in our work and elsewhere<sup>5,7,8/</sup> that carbon monoxide is commonly found when hot tungsten filament devices are used. Helium is present due to permeation from the atmosphere through the warm glass envelope of the ion gauge. Because helium is not pumped well by the ion gauge,

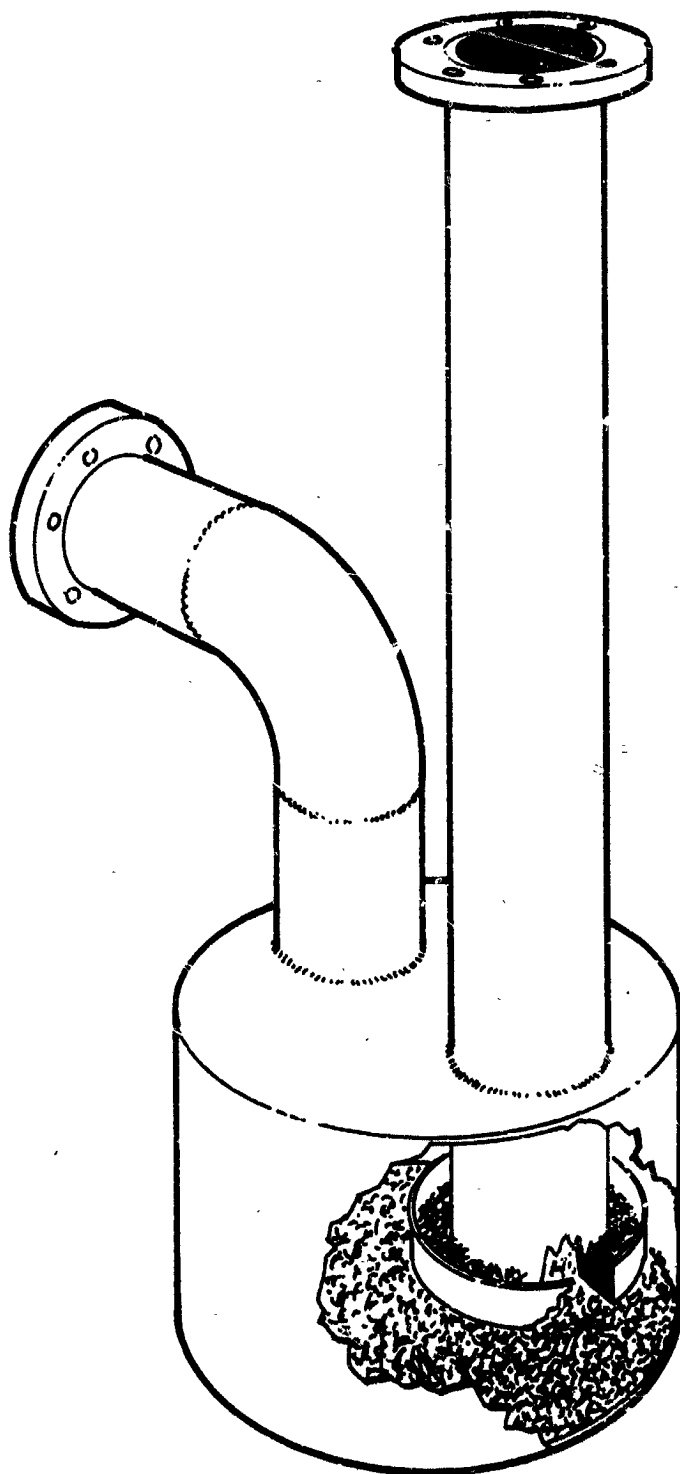


Fig. 2 - Special Chemical Trap in Which the Trapping Zone Can Be Submerged into Cryogenic Fluids. Trap is constructed of heli-arc welded 304 stainless steel. Chemicals are placed in lower part of sump and in annular tray at base of vertical tube.

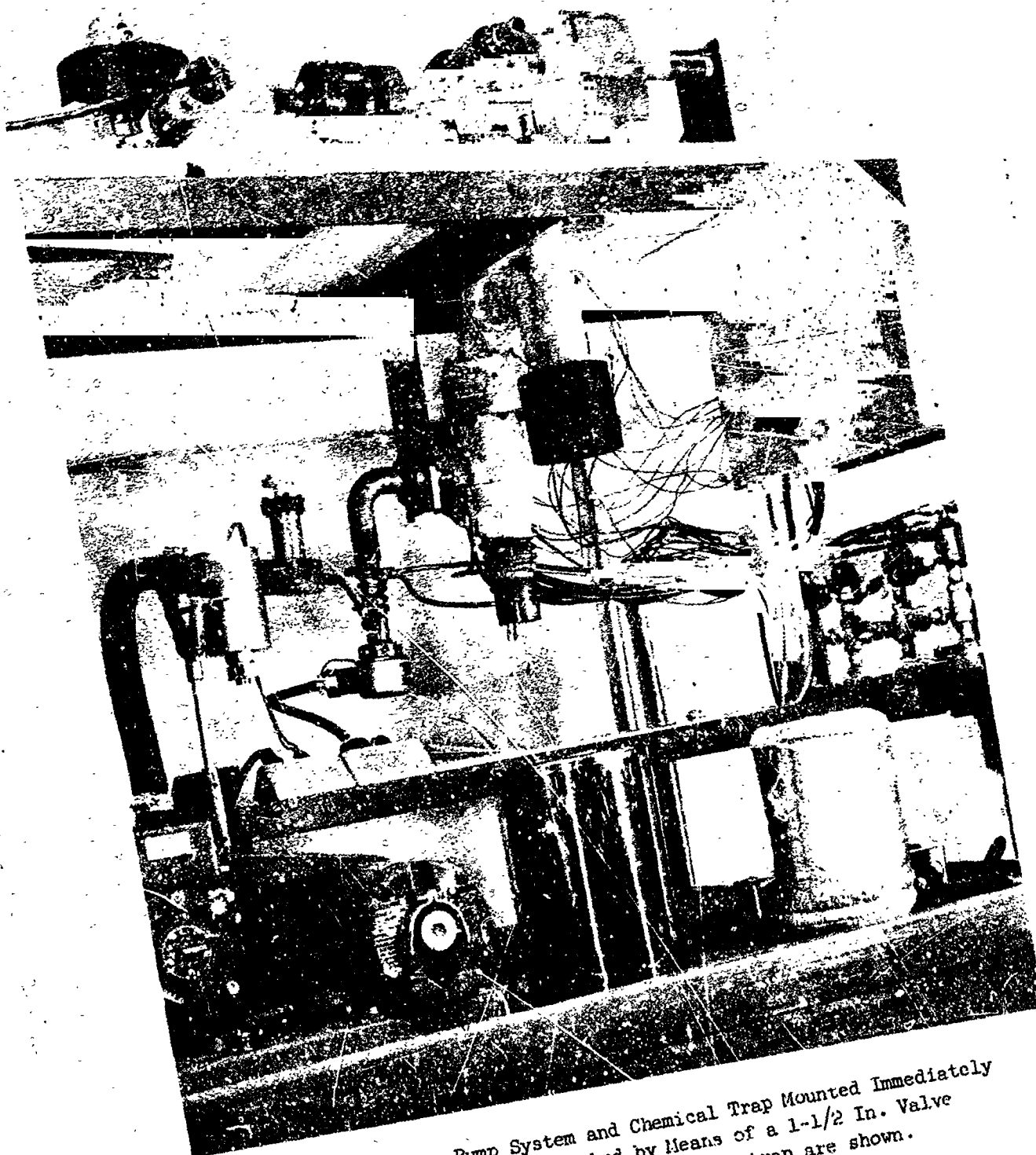


Fig. 3 - Oil Diffusion Pump System and Chemical Trap Mounted Immediately Below the PPAS and Attached by Means of a 1-1/2 In. Valve Dewar and heating mantle for chemical trap are shown.

TABLE I

RESIDUAL GASES IN AN OIL DIFFUSION PUMPED UHV SYSTEM FOR SEVERAL TRAPPING  
CONDITIONS

GAS	BACK GROUND	CHEMICAL IN TRAP			NO CHEMICAL IN TRAP		
		-196°C	-78°C	25°C	-196°C	-78°C	25°C
H <sub>2</sub>	1.3 x 10 <sup>-10</sup>	2.1 x 10 <sup>-9</sup>	2.1 x 10 <sup>-9</sup>	1.8 x 10 <sup>-9</sup>	3.1 x 10 <sup>-9</sup>	2.5 x 10 <sup>-9</sup>	2.2 x 10 <sup>-9</sup>
He	5.1 x 10 <sup>-11</sup>	—	—	—	—	—	—
CH <sub>4</sub>	2.2 x 10 <sup>-10</sup>	1.7 x 10 <sup>-11</sup>	6.8 x 10 <sup>-11</sup>	7.1 x 10 <sup>-11</sup>	1.6 x 10 <sup>-11</sup>	8.3 x 10 <sup>-11</sup>	1.3 x 10 <sup>-10</sup>
H <sub>2</sub> O	1.6 x 10 <sup>-12</sup>	8.4 x 10 <sup>-12</sup>	8.7 x 10 <sup>-12</sup>	7.3 x 10 <sup>-12</sup>	9.4 x 10 <sup>-12</sup>	5.0 x 10 <sup>-12</sup>	6.3 x 10 <sup>-12</sup>
C <sub>2</sub> H <sub>2</sub>	—	—	—	—	—	—	1.3 x 10 <sup>-12</sup>
C <sub>2</sub> H <sub>4</sub>	5.0 x 10 <sup>-12</sup>	1.1 x 10 <sup>-12</sup>	4.8 x 10 <sup>-12</sup>	3.3 x 10 <sup>-11</sup>	6.1 x 10 <sup>-12</sup>	8.8 x 10 <sup>-11</sup>	1.2 x 10 <sup>-10</sup>
CO	7.8 x 10 <sup>-11</sup>	1.5 x 10 <sup>-11</sup>	2.1 x 10 <sup>-11</sup>	5.7 x 10 <sup>-11</sup>	2.1 x 10 <sup>-11</sup>	1.3 x 10 <sup>-10</sup>	2.0 x 10 <sup>-10</sup>
C <sub>2</sub> H <sub>6</sub>	3.4 x 10 <sup>-12</sup>	1.3 x 10 <sup>-12</sup>	8.2 x 10 <sup>-12</sup>	9.5 x 10 <sup>-12</sup>	1.2 x 10 <sup>-12</sup>	7.3 x 10 <sup>-12</sup>	1.9 x 10 <sup>-11</sup>
A	5.6 x 10 <sup>-13</sup>	—	—	—	—	—	—
C <sub>3</sub> H <sub>6</sub>	—	6.3 x 10 <sup>-13</sup>	7.2 x 10 <sup>-13</sup>	2.1 x 10 <sup>-12</sup>	4.7 x 10 <sup>-13</sup>	2.2 x 10 <sup>-12</sup>	4.6 x 10 <sup>-12</sup>
CO <sub>2</sub>	2.6 x 10 <sup>-12</sup>	2.1 x 10 <sup>-12</sup>	2.4 x 10 <sup>-12</sup>	3.6 x 10 <sup>-12</sup>	2.6 x 10 <sup>-12</sup>	3.8 x 10 <sup>-12</sup>	4.5 x 10 <sup>-12</sup>
C <sub>4</sub> H <sub>8</sub>	—	—	—	—	3.6 x 10 <sup>-13</sup>	8.5 x 10 <sup>-13</sup>	2.3 x 10 <sup>-12</sup>
C <sub>6</sub> H <sub>6</sub>	—	8.4 x 10 <sup>-14</sup>	1.8 x 10 <sup>-12</sup>	1.1 x 10 <sup>-13</sup>	6.4 x 10 <sup>-13</sup>	6.3 x 10 <sup>-10</sup>	5.3 x 10 <sup>-10</sup>
C <sub>7</sub> H <sub>8</sub>	—	—	—	—	—	—	2.3 x 10 <sup>-12</sup>
C <sub>8</sub> H <sub>10</sub>	—	—	—	—	—	3.0 x 10 <sup>-13</sup>	9.1 x 10 <sup>-13</sup>
TOTAL	4.8 x 10 <sup>-10</sup>	2.1 x 10 <sup>-9</sup>	2.2 x 10 <sup>-9</sup>	2.1 x 10 <sup>-9</sup>	3.2 x 10 <sup>-9</sup>	3.8 x 10 <sup>-9</sup>	3.3 x 10 <sup>-9</sup>

the partial pressure of this gas gradually increases with time. When either the getter ion pump or diffusion pump is operating on the PPAS no detectable amount of helium is observed. The remainder of the background spectrum indicates relatively small amounts of water, ethene, ethane, argon and carbon dioxide.

The diffusion pump data\* are displayed in Table I under two major groups, i.e., "chemical in trap" and "no chemical in trap." Each group is divided into three sections corresponding to the trap operating conditions, i.e., submerged in liquid nitrogen, or a dry ice and acetone slurry, or at ambient laboratory conditions.

Scanning of the diffusion pump data indicates that the major gas species in all cases is hydrogen. The relatively large amounts of hydrogen are due to desorption from metal parts in the diffusion pump and Chevron baffle which were not baked at high temperature. The absence of helium and argon is explained above.

The objective of this study was to determine the level of contamination in an oil diffusion pumped UHV system and to explore methods which would be practical and effective in further reducing the problem of oil contamination. Therefore, it is necessary to establish a criteria for determining contaminating effects.

A review of the hydrocarbon gas species in Table I clearly indicates that benzene ( $C_6H_6$ ) is the species which shows the greatest dependence upon trap temperature. This is not an unexpected observation<sup>9,3/</sup> since the chemical composition of DC-705 is pentaphenyltrimethyltrisiloxane.<sup>10/</sup> The generation of phenyl groups would be expected due to cracking of the fluid in the boiler of the diffusion pump. The additional hydrogen needed to form benzene would then be available to the phenyl group during their interactions with the metal wall of the system. It was interesting to note that in this investigation no detectable amount of methyl or phenyl has been observed except for expected amounts which appear in fragmentation patterns for hydrocarbons in the system. The heavier hydrocarbons were methylated benzenes. Therefore, based on the above references and the data collected in this study, it will be assumed that the amount of benzene (gas) in the system is most representative of the presence of oil contamination in the pressure range  $<10^{-8}$  torr.

\* Getter ion pump is isolated from system.

Based on the above assumption it is interesting to examine the relative changes in the amount of benzene present for the various trapping conditions.\* The best operating condition is observed for the trap with chemicals at liquid nitrogen temperature. The presence of chemicals reduces the contamination by a factor of  $\sim 8$  in comparison to the nonchemical situation. However, the most outstanding effect is noted for the  $-78^\circ\text{C}$  condition. At this temperature chemicals are responsible for reducing contamination by a factor of  $\sim 350$ . Note also that the difference between the  $-78^\circ\text{C}$  chemical condition and the  $-196^\circ\text{C}$  nonchemical condition is only by a factor of  $\sim 3$ . The reduction of benzene due to chemicals for ambient laboratory temperature is by a factor of  $\sim 5$ . Ethene also exhibited a trap temperature dependence similar to benzene although not nearly as intense.

The partial pressure of methane did not change greatly (less than an order of magnitude) for various trapping conditions. However, the methane pattern repeatedly increased to the background level upon isolating the PPAS from the trap and diffusion pump. When the valve to the getter ion pump was opened, the pressure of methane would fall to  $\sim 1.6 \times 10^{-11}$  torr. This behavior indicated that there was a source of methane in the PPAS.

At this point it is interesting to refer for a moment to another study which was conducted in this laboratory. This work involved determining the major residual gas constituents in a liquid nitrogen trapped mercury diffusion pumped glass and metal system. The analyzer used in this study was similar to that described above. Table II displays the residual gases following a bake of  $200^\circ\text{C}$ . The total pressure measurement was made with a Redhead gauge.

TABLE II

MAJOR RESIDUAL GASES IN  $\text{LN}_2$  TRAPPED MERCURY DIFFUSION PUMPED  
GLASS-METAL UHV SYSTEM

$\text{H}_2$	$1.7 \times 10^{-11}$ torr (air equivalent units)
$\text{H}_2\text{O}$	$1.8 \times 10^{-11}$
CO	$1.5 \times 10^{-10}$
$\text{CO}_2$	$1.7 \times 10^{-11}$
Total	$2.0 \times 10^{-10}$

Note that no detectable amount of methane was observed. This fact indicates that the analyzer itself is not generating significant amounts of methane.

\* Conditions are established for approximately five hours before data is taken.

The generation of methane from Bayard-Alpert type gauges with metal envelopes has been reported by Davis.<sup>E</sup> However, in the PPAS the ionization gauges are mounted in glass envelopes. In order to explore further the possibility of the ion gauge being a methane source, a brief study was conducted to determine the effects of a hot tungsten filament and the bombardment of a metal surface by electrons on the residual gas in the PPAS. The operating conditions of the ionization gauges (shown in Fig. 1) were varied according to the following list

1. Both filaments off;
2. One filament on only;
3. One filament on with +130 V grid potential; and
4. One filament on with +350 V grid potential.

It was determined that when the oil diffusion pumped system was operating with a liquid nitrogen cooled chemical trap, the amount of methane present is not affected greatly by the presence of a hot filament but is very sensitive to the energy with which electrons bombard metal gauge parts (grid). This observation indicates that methane resided in the system as an adsorbed species which further suggests that the amount of methane contamination in a well baked glass metal system is a function of the history of the system or pumping technique rather than the presence of a hot tungsten filament. It was also observed that the methane level was dependent on the operation of a hot filament when the study was conducted before baking the system.

The remaining gases in Table I do not appear to be greatly dependent upon the experimental trap temperature. However, there is a definite advantage indicated in the use of chemicals for reducing hydrocarbon contamination.

#### D. Summary

A systematic study has been conducted to determine the effectiveness of an optically dense chemical trap in reducing the gaseous contamination of UHV systems by an oil diffusion pump using DC-705 pumping fluid. Three trap temperature conditions were studied. Two of these conditions were determined by submerging the experimental trap into a bath of either liquid nitrogen or dry ice and acetone. The third temperature was determined by ambient laboratory conditions.

The data collected by means of a sensitive partial pressure analyzer indicated that a significantly lower level of contamination was achieved by cooling the chemical trap to even -78°C from room temperature. Data were also

taken of the trap operating without chemical. Comparison of these sets of data indicates that a significant increase in trapping efficiency can be realized when chemicals are used. The greatest improvement over the nonchemical conditions was noted at the dry ice and acetone slurry temperature. A consideration of these results suggests that reasonably good trapping of UHV systems can be achieved by employing mechanically refrigerated chemical traps above oil diffusion pumps.



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